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Studies of free compound semiconductor surfaces as well as gas-solid and metal-solid interaction are summarized. An experimental and theoretical analysis of the GaAs(110) surface reconstruction was conducted and applied to three additional compounds: InSb, InP and ZnTe. Attempts were made to relate the type and the magnitude of the surface reconstruction of these materials to the covalent-ionic character of the bonding. Knowledge of the atomic reconstructions of these

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surfaces was used to investigate the reactivity of semiconductor surfaces (GaAs(110)) with gas(oxygen)and metal(aluminum) atoms. These multicomponent systems are at the center of the author's essays to understand the chemistry of these surfaces and its relationship with their atomic reconstruction. Using Low Energy Electron Diffraction (LEED) as the principal tool of investigation, structural information has been obtained which correlates chemical and electronic information obtained by other surface analysis techniques.

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During the past three years, and under the support of the Army Research Office, we have increased our effort in the study of free compound semiconductor surfaces as well as gas-solid and metal-solid interaction.

We have first refined our method of surface structure determination for semiconductor compounds. We completed, both experimentally and theoretically, the analysis of the GaAs(110) surface reconstruction and applied this technique to three additional compounds: InSb, InP and ZnTe. We have tried to relate the type and the magnitude of the surface reconstruction of these materials to the covalent-ionic character of the bonding. Results are reviewed below. We have then used the necessary knowledge of the atomic reconstructions of these surfaces to investigate the reactivity of semiconductor surfaces (GaAs(110)) with gas ( $O_2$ ) and metal (Al) atoms. These multicomponent systems are at the center of our essays to understand the chemistry of these surfaces and its relationship with their atomic reconstruction. Using Low Energy Electron Diffraction (LEED) as the principal tool of investigation, we have been able to provide structural information which correlate chemical and electronic information obtained by other surface analysis techniques.

#### 1) The Atomic Structure of Free Semiconductor Surfaces

We focused our LEED study on the (110) faces of four zincblende materials - GaAs, InSb, ZnTe and InP - in order to try to establish how various physical and chemical properties such as bulk crystallographic structure, bonding ionicity, band gap and surface index, affect their

surface atomic reconstruction. As an example, the ionicity of these materials, on the Phillips scale,<sup>1</sup> range from very covalent with GaAs (.310) and InSb(0.321) to more ionic with InP(0.421) to very ionic with a II-VI compound, ZnTe(0.546).

#### GaAs(110)

The selvage structure proposed by our single scattering analysis<sup>2,3</sup> has been refined by a detailed multiple scattering computation<sup>4</sup> for which a sensitivity analysis was performed. The output lead first to the selection of the most probable surface structure for GaAs(110) as one in which the top layer undergoes both a rigid rotation of  $27.4^\circ$  and a  $0.05\text{\AA}$  contraction with the As atoms moving outward and the Ga atoms inward, giving a relative vertical shear of  $0.65\text{\AA}$ . In the second layer, the Ga moves outward  $0.06\text{\AA}$  and the second layer As moves inward  $0.06\text{\AA}$ . There is no evidence for third-layer distortions. It also confirmed the validity of our single scattering analysis as a means of narrowing very efficiently the range of atomic positions. We have now adopted, in our surface structure search, a systematic cycle of computation: the single scattering (kinematic) computation is used to produce a structure which lies close to the real surface structure of the crystal, and the more expensive but precise multiple scattering (dynamical) computation is then used to refine the structure to an optimum output.

The important features of the GaAs(110) reconstruction are that atomic displacements penetrate two layers into the substrate. The reason for this, we believe, is that GaAs is among the more covalent of the III-V compounds so that bond length conservation is a strong tendency in this

material. The terminating As-sublattice is displaced outward from its ideal terminating position because of rehybridization from the bulk  $sp^3$  configuration to the more pure p-like configuration stemming from breaking of the surface bond. This has the effect of reducing the bond angle to the nearest neighbor atoms, hence the upward displacement of the As-sublattice. By the same token, the Ga sublattice is displaced inward because it tends to rehybridize to an  $sp^2$  configuration with nearly planar bonding. The net result is that the sublattices of the terminating plane tend to assume some compromise between their lattice bonding configuration and the bonding configuration they might have in small molecules. Because of the bond-length conservation tendency, a strain field is set up in selvage region. It is the relaxation of this strain field that causes the subsurface atomic displacements. The optimized structure is summarized in Table I for which all necessary parameters are defined in Fig. 1.

#### InSb(110)

For two specific reasons, this material was expected to behave like GaAs. Its ionicity (0.321) is very close to the one of GaAs(0.310), and its atomic constituents separated by only one element on the periodic table. Their masses are very similar and so are their atomic scattering factors.<sup>5</sup> The thermal atomic vibrations of the two atomic species are therefore virtually identical which considerably simplifies the problem of structure determination. Using the same combination of kinematical and dynamical model calculations, we determined the surface reconstruction which, as expected, was found to be very similar to that of GaAs(110).

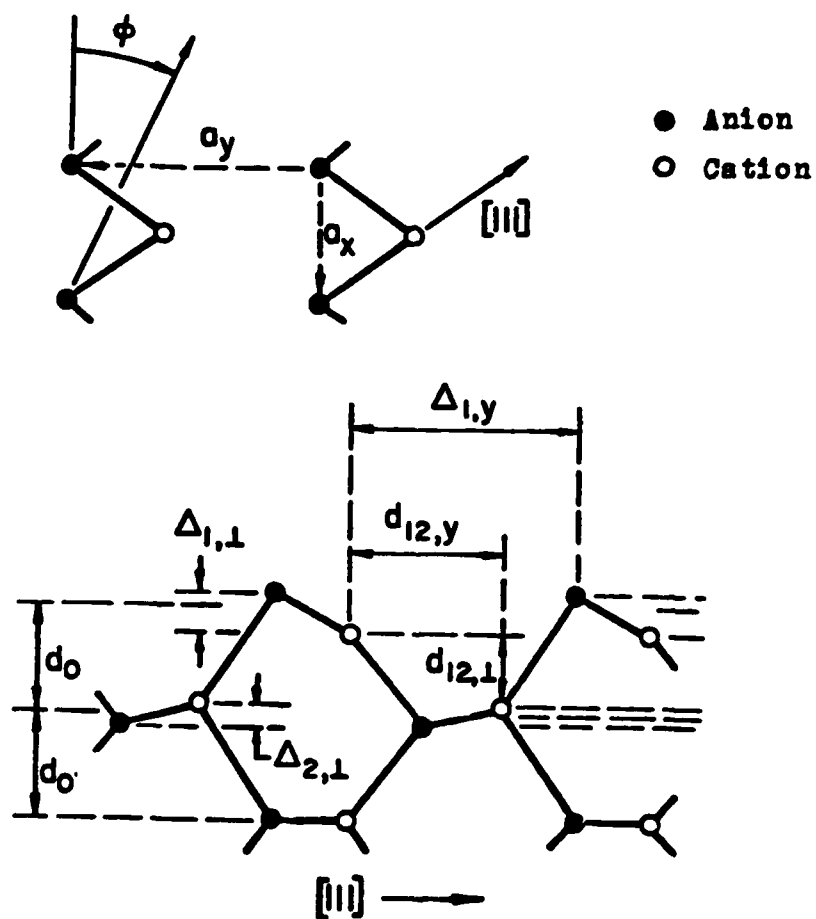


Fig. 1 Elevation of the three first atomic layers of the zincblende lattice showing the distortions of the first and second layers.



Material	Phillips Ionicity	Lattice Constant	Layer	Vertical Anion Displacement	Vertical Cation Displacement	Horizontal Anion Displacement	Horizontal Cation Displacement	$\Delta_{1,1}$	$\Delta_{2,1}$	$\theta_1$
GaAs(110)	0.310	5.654	1	+0.144	+0.506	-0.332	-0.486	0.650	-0.120	27.34°
			2	+0.06	+0.06	0.0	0.0			
			3	0.0	0.0	0.0	0.0			
InSb(110)	0.321	6.478	1	+0.177	+0.604	-0.384	-0.584	0.781	-0.180	28.8°
			2	+0.090	+0.090	0.0	0.0			
			3	0.0	0.0	0.0	0.0			
InP(110)	0.421	5.869	1	+0.060	+0.630	-0.344	-0.516	0.690	-0.130	28.05°
			2	+0.065	+0.065	0.0	0.0			
			3	0.0	0.0	0.0	0.0			
ZnTe(110)	0.546	6.089	1	+0.200	+0.550	-0.182	-0.560	0.750	0.0	33.25°
			2	0.0	0.0	0.0	0.0			
			3	0.0	0.0	0.0	0.0			

Table I

The atomic displacements in the InSb(110) selvage differ only quantitatively from those of the GaAs(110) selvage.<sup>6</sup> Most important is the fact that atomic displacements also occur up to two layers into the substrate in the InSb(110) selvage, presumably because of the strongly covalent character of the material which tends to conserve bond lengths, establish strain fields in subsurface layers and generate subsurface atomic displacements through the relaxation of these strain fields. The details of the reconstruction are given in Table I.

#### ZnTe(110)

This is a II-VI compound of significant bonding ionicity. Furthermore, it is a truly diatomic material for LEED in the sense that the atomic mass of Te is about twice the one of Zn. Their scattering factors are equally different.<sup>7</sup>

The ZnTe(110) surface proved to be one of the most difficult from which to obtain a structure because the various kinematical approaches are not as successful as in the case of GaAs and InSb. The two sublattices contribute in different ranges of primary energy in greatly differing degrees. This is not the case for the "quasi-monatomic" lattices because these contributions are strongly convoluted. In addition, the thermal atomic vibrations of Zn and Te could not be included through a simple Debye-Waller factor and a surface Debye temperature. The two sublattices vibrate rather differently and there is little guidance in the published literature as to how to include lattice vibrations into the model calculations.

We were able to establish why the typical compound semiconductor whose sublattice atomic masses are substantially different will not lend themselves as easily to selvage structure analysis as the less conventional

but technological important material GaAs and InSb. Our analysis lead to the selection of the most probable surface structure for ZnTe(110) as one in which the top layer Te moves outward  $0.20\text{\AA}$  and the top layer Zn moves inward  $0.55\text{\AA}$ . No convincing evidence is obtained for second layer reconstruction.<sup>8</sup> Rather than attempting to maintain constant bond lengths and the associated strain fields that generate subsurface atomic displacements in the more covalent materials, the more ionic materials respond differently to the breaking of the surface bonds. The Madelung potential at the surface is slightly reduced,<sup>9</sup> which, in turn, influences the amount of charge transfer and the ionic radii of the surface atoms, and a change in ionic radii is the equivalent of a bond length change. Thus, with the more ionic materials, the surface bond lengths are affected by the generation of a free surface, and much less of a strain field is established to cause subsurface displacements. The details of the reconstruction can be found in Table I.

#### InP(110)

Technologically more and more important, InP also presents characteristics that make it interesting in a general study of semiconductor surfaces.

(1) Its ionicity ( $0.421$ )<sup>1</sup> lies between those of GaAs and InSb on the covalent side and ZnTe on the ionic side. (2) The atomic masses of the constituents are very different and so are their scattering factors. (3) The surface composition is very sensitive to the details of the preparation method: cleaving or sputtering-annealing. The analysis of the LEED data obtained from this material has been completed very recently and will probably be the object of further refinements. Nevertheless, the details of the atomic structure are reported in Table I. We also verified the

similarity between the LEED intensity profiles taken from cleaved and sputtered-annealed surfaces,<sup>10</sup> thus confirming the results obtained for GaAs(110).<sup>11</sup> This result will most probably be verified on all (110) faces of zincblende compound semiconductors.

In summary, the III-V and II-VI compounds analyzed so far have all shown surface atomic geometry distorted from that of a truncated bulk solid, although the space group symmetries at the surface are indistinguishable from those of the corresponding bulk materials. The surface reconstruction involve large ( $\sim 0.5\text{\AA}$ ) displacements of the species in the uppermost atomic layer and often smaller displacements in the layer beneath. The character of these reconstructions depends, however, on the nature of the chemical bonding in the bulk materials. For the most covalent materials, i.e., GaAs and InSb, the anion rotates outward and the cation inward in a fashion such that bond lengths remain unaltered to within 1% except for a 5% contraction of the bond between the uppermost anion and the cation in the layer beneath. Moreover, these "covalent" reconstructions penetrate at least two atomic layers into the surface with the displacements in the second layer being in the opposite sense to those in first. The surface reconstructions of the more ionic materials, i.e., ZnTe and ZnO, are characterized by generally smaller displacements of the anion species and the absence of definitive evidence for distortions of the second and deeper layers from the bulk geometry. Therefore available structural data suggest a systematic change in the nature of the surface reconstructions of the cleavage faces of tetrahedrally-coordinated compound semiconductors with increasing ionicity. This change is most pronounced for materials with

ionicities in the vicinity of those of ZnTe and InP, and seems to be associated with the transition from III-V materials (which exhibit ionicities  $f_1 \leq 0.5$ ) to II-VI materials (which exhibit ionicities  $f_1 \geq 0.5$ ).

## 2) The Gas-Solid Interaction: Oxygen on GaAs(110)

In order to help resolve the issue of the nature of the interaction between oxygen and GaAs, it became most important to use LEED, a structure sensitive probe to complete the abundant set of experimental (XPS, UPS)<sup>14,16,17</sup> and theoretical (small cluster quantum chemical computation)<sup>15</sup> data dealing with oxygen on GaAs(110). A definite uncertainty on the nature of the interaction had resulted from the previous studies.

The good knowledge we had of the structure of the clean surface of GaAs was the ideal starting point for such a study. Having intimately related, in our previous work on GaAs, the different aspects of the intensity profiles of the diffracted beams to the structural characteristics of the free surface (e.g. the 129 eV and 162 eV peaks of the  $(\bar{1}\bar{1})$  beam linked directly to the reconstruction of the second layer of the crystal),<sup>3</sup> it became most informative to monitor closely the intensity profiles obtained from the surface as a function of the exposure to oxygen.

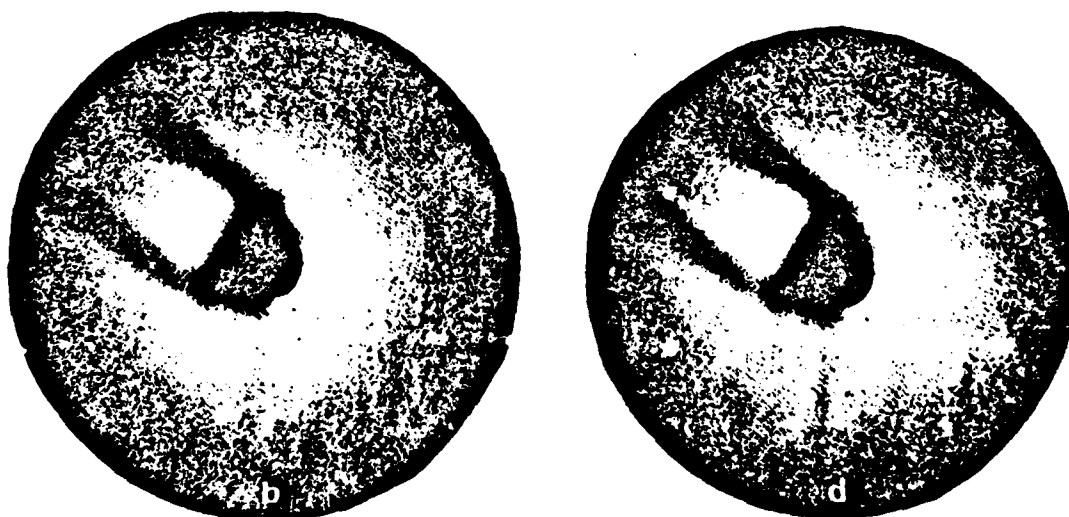
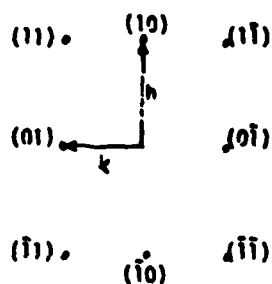
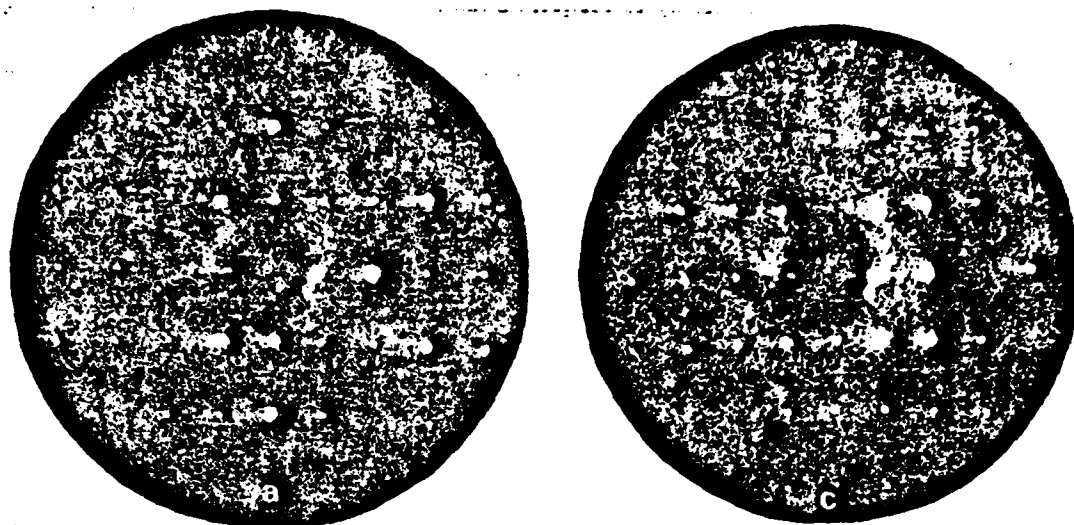
The main result is that the interaction is of a disordering nature.<sup>12,13</sup> We took LEED measurements for a very large range of exposure to molecular oxygen ( $10^3 - 10^{14}$  L) and at no time did we observe any evidence for an ordered oxygen chemisorption on the surface. Between  $10^3$  L and  $10^8$  L, the diffraction pattern progressively weakens due to the increase of the disordered patch areas on the surface. At  $5 \cdot 10^8$  L, the surface layer is entirely disordered: a thin (1-3 Å) layer covers the GaAs lattice which no longer

shows any evidence of the atomic displacements observed on the free surface. The resulting pattern presents a high background due to the incoherent scattering in the amorphous layer and very sharp diffraction spots due to electrons diffracting coherently in the substrate and traversing the thin amorphous layer (Fig. 2). The intensity profiles are considerably modified with respect to the "clean surface profiles", they present the characteristics of a bulk-like diffraction.

When the oxygen exposure is increased, the structure is not further modified. The amorphous layer now protects the lattice and no depth oxidation is observed, at least up to an exposure of  $10^{14}$  L (40 hours at 1 atm. of oxygen).

The experiment was then performed with excited oxygen (the hot ion gauge filament was kept on during the experiment). The end result was identical as far as LEED is concerned. Only the reaction rate was faster (100 times). We can summarize these results as follows:

- a. There is no evidence of ordered chemisorption of oxygen on GaAs(110) at room temperature.
- b. At  $5 \cdot 10^8$  L of unexcited oxygen, a complete thin ( $1-3\overset{\circ}{\text{Å}}$ ) amorphous layer covers the lattice. This amorphous layer can be due to: (1) the formation of surface oxides ( $\text{Ga}_2\text{O}_3, \text{As}_2\text{O}_3$ ) and (2) an oxygen chemisorption disordered by the added induced strain on the geometry of the surface.
- c. The rate of the reaction increases when excited oxygen is used, which seems to support the concept of a dissociative, defect-induced oxidation of the surface.



Photographs of the LEED pattern from the clean (a) and oxidized (b) surface at 150 eV. The  $(hk)$   $(hk)$  symmetry is very clear. The diffraction spots are very sharp. The  $(0\bar{1})$  peak, large in (a) is reduced to zero in (b). The  $(\bar{1}0)$  beam disappears in (b). Same disposition for (c) and (d) taken at 168 eV. The  $(\bar{1}\bar{1})$  peak, bright in (c), disappears in (d) (see fig. 3). Same observation for the  $(\bar{1}\bar{2})$  and  $(0\bar{2})$  beams.

Figure 2

### 3) Metal Overlayers: Al on GaAs(110)

As theoretical computations of chemical and electrical properties (Schottky barriers) of metal-semiconductor interfaces are developed, a detailed knowledge of the geometry of these interfaces becomes necessary: changes in chemical bonds and charge redistribution are intimately related to the microscopic geometry of the compound. In the case of Al on GaAs, it is believed that the surface layer undergoes a reaction through which the Al replaces the Ga. Arguments in favor of this reaction are given by measured photoemission peak shifts:<sup>18,19</sup> the Ga 3-d core level shifts toward lower binding energy (0.95 eV) indicating the possibility of having nearly free Ga atoms on the surface, while the Al 2-p level shift toward higher binding energy (0.7 eV) indicating that Al is reacted as AlAs. We have started to analyse LEED data<sup>20</sup> collected from GaAs surfaces covered with 0.5 monolayer (ML), 0.75ML, 1ML, 1.5ML of Al and which can be described as follows.

After a room temperature deposition, the Al overlayer does not produce any change in the shape of the LEED profiles aside from a general lowering of their intensity, indicating that the Al is disordered, probably randomly chemisorbed on the surface. At that stage of the study, we do not believe that most Al atoms can be involved in a replacement reaction. LEED calculations show that, under such conditions, the intensity profiles would present changes, in contradiction with the experimental results.

After a 30mn, 450°C annealing cycle, very definite and reproducible changes can be observed in the intensity profiles, indicating the presence of a new ordered structure at the surface of the crystal. The thermal energy provided to the system has ordered the Al overlayer which now lies in



registry with the substrate lattice. The LEED pattern conserves its  $(hk)-(\bar{h}\bar{k})$  symmetry which implies that the Al atom occupies a symmetrical site in the unit cell.

LEED measurements taken for progressively thick layers of Al show the continuous character of these changes: there are no specific patterns or profiles corresponding to 0.5ML, 1ML, 1.5ML and more. This feature seems to point out the progressive formation of a new compound at the surface, AlAs, resulting from the Al-Ga replacement reaction. However, this structural study is still in an early stage and more experimental data are being collected. In order to reduce the uncertainty, chemisorption versus Al-reaction and formation of AlAs, more LEED and photoemission work must be done. A careful measurement of LEED profiles for larger  $(2-10\text{\AA})$  thicknesses must be performed. Theoretical LEED computations involving both chemisorbed overlayers and an increasing number of AlAs layers on top of the GaAs substrate must be performed. Finally, the photoemission work (UPS, XPS) mentioned above must be repeated on the annealed Al overlayer in order to correlate chemical and structural information.

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